

$$|[(Ts)-(Tr)]-[(Ts)-(Tr)]|$$

This can also be understood from the fact that the algebraic difference between the lengths of two sides of the triangle is different from the length of the other one side.

In summary, the instrument disclosed in U.S. Pat. No. 5,224,775 is also unsuccessful in extracting the heat capacity component of the DSC signal by the instrument itself.

### SUMMARY OF THE INVENTION

It is an object of the invention to provide an instrument for automatically separating the heat capacity component of a signal and the latent heat component without relying on human intervention, which would have been impossible to achieve by the prior art DSC instrument.

To achieve the above and other objects of the invention, and to solve the above problems, the present invention uses the structure of a conduction type calorimeter. In particular, the heat flow between an unknown sample and a heat reservoir is found from the temperature difference between two points along a heat flow path going from the heat reservoir to the unknown sample. Independently of this, the heat flow between a reference sample and the heat reservoir is found from the temperature difference between two points along a heat flow path going from the heat reservoir to the reference sample. The temperature of the heat reservoir is controlled by a control signal which varies in time according to a ramp function which is modulated by an AC function, in the same way as in U.S. Pat. No. 5,224,775. A heat flow signal on the side of the unknown sample and a heat flow signal on the side of the reference sample are independently demodulated and their respective heat flow amplitudes are found. Then, the difference between them is taken to obtain the amplitude of an excessive heat flow supplied to the unknown sample. This excessive heat flow amplitude is divided by a sample temperature amplitude and an AC angular frequency which are found independently of the excessive heat flow amplitude. Thus, the excessive heat capacity of the unknown sample with respect to the reference sample is obtained, in the same manner as in an AC calorimeter. Furthermore, the excessive heat capacity of the unknown sample is multiplied by the average temperature change rate of the unknown sample so that the excessive heat capacity may be transformed into units of heat flow. This is produced as a heat capacity component signal. Although a DSC signal contains both information concerning the heat capacity of an unknown sample and information concerning the heat capacity, the heat capacity component signal reflects only the former component and represents the baseline of the DSC signal. The heat flow signal on the side of the unknown sample is passed through a low-pass filter. The heat flow signal on the side of the reference sample is processed similarly to derive a low-frequency signal. This low-frequency signal is subtracted from the low-frequency signal from the aforementioned low-pass filter. As a result, a kinetic component signal containing information regarding the latent heat of the unknown sample is obtained.

The structure described above operates in such a way that the instrument itself separates and extracts information concerning the heat capacity of the unknown sample and information concerning the latent heat. The former information has been conventionally unavoidably included in the DSC signal. The latent heat is produced when the unknown sample causes or undergoes a transition or reaction. The resulting heat capacity component signal indicates the position of the correct baseline about the original DSC data.

Since the kinetic component signal does not change when the heat capacity of the unknown sample changes, information about enthalpy produced during a transition or reaction can be known precisely.

### BRIEF DESCRIPTION OF DRAWING

FIG. 1 is partly an elevational cross-sectional view and partly a block circuit diagram illustrating one embodiment of a thermal analysis instrument according to the invention.

FIG. 2 is a diagram illustrating the operating principle of a prior art instrument.

### DESCRIPTION OF THE PREFERRED EMBODIMENTS

One embodiment of the present invention is described in detail below with reference to the drawing.

In FIG. 1, a heat reservoir 1 having a substantially H-shaped cross section in a vertical plane is made of, e.g., silver. The temperature ( $T_h$ ) within heat reservoir 1 is measured by a furnace temperature-measuring thermocouple 2. The signal from the thermocouple is fed to a furnace temperature control circuit 3, which in turn supplies electric power to a heater 4 which surrounds reservoir 1 and is enclosed in an insulator. In this way, the temperature of heat reservoir 1 is controlled. For this purpose, a well-known PID (proportional plus integral plus derivative) control action is utilized. In particular, the difference between a temperature indicated by a desired temperature program delivered from a processor 16 and the output temperature from the furnace temperature-measuring thermocouple 2 is produced and subjected to PID operations in the furnace temperature control circuit 3. Output power from the control circuit 3 is supplied to the heater 4.

The desired temperature information produced by processor 16 is dependent on signals provided by setting a constant rate temperature-varying means 20 and an alternating temperature-varying means 21, both connected to processor 16. Appropriate values are set into means 20 and 21 by an operator via a suitable input device, such as a keyboard. Means 20 are set to provide a signal representing a desired constant rate of temperature variation, and means 21 are set to provide an alternating signal representing a desired frequency and peak amplitude of an alternating temperature component which will be superimposed on, or modulate, the constant rate signal.

A heat conduction plate 6 made of constantan (copper-nickel alloy) is mounted at the center of heat reservoir 1 such that the center of plate 6 is fixed inside heat reservoir 1. An unknown sample support portion 6a is formed like a platform at one end of heat conduction plate 6. A reference sample support portion 6b is formed at the other end of heat conduction plate 6. Thus, these two portions 6a and 6b are arranged symmetrically to one another and to the center of heat reservoir 1. An unknown sample loaded in an aluminum container 7 is placed on the unknown sample support portion 6a. A hollow container 7 is placed on the reference sample support portion 6b.

A cover 5 made of, e.g., silver is mounted at the top of heat reservoir 1 to close heat reservoir during operation and is removable to permit containers 7 to be inserted and withdrawn. A chromel-alumel thermocouple 9 (of the K type) comprising a chromel line 9a forming a positive pole and an alumel line 9b forming a negative pole is welded to the underside of the unknown sample support portion 6a to measure the temperature of the unknown sample. Also, a